DENTAL MATERIALS 36 (2020) 1183–1189

Available online at www.sciencedirect.com

ScienceDirect

journal homepage: www.intl.elsevierhealth.com/journals/dema

Characterization of heat resistant hydraulic sealer for warm vertical obturation

*Mohammed Hadis, Josette Camilleri**

School of Dentistry, Institute of Clinical Sciences, College of Medical and Dental Sciences, University of Birmingham, Birmingham, United Kingdom

ARTICLE INFO

Article history:
Received 30 March 2020
Accepted 18 May 2020

Keywords:
Root canal sealers
Warm vertical compaction
Sealer characterization

ABSTRACT

Objective. Warm vertical compaction of gutta-percha is a technique that is used by most specialists for root canal obturation. The sealers currently available exhibit irriversible chemical changes when heated. New biologically active sealers that do not sustain irreversible changes when heated are an attractive alternative to be used with warm vertical compaction obturation technique. The aim of this study was to measure the heat generated by warm vertical compactors inside the root canal, characterize a newly developed root canal sealer at different temperatures and verify its suitability at the actual temperature window used clinically.

Methods. The typical temperatures generated by two heat carriers in a root canal were assessed by thermocouples. Two premixed root canal sealers TotalFill BC and HiFlow BC (FKG, Switzerland) were allowed to set and they were characterized by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction analysis (XRD) immediately after setting and by XRD after 28 days in physiological solution. The ion leaching in solution was assessed by inductively coupled plasma (ICP). The organic component was extracted in acetone and assessed by Fourier transform infrared spectroscopy (FT-IR) for both the unset sealer and sealer subjected to different temperatures. The heat profiles of both sealers were investigated by FT-IR and thermographic analysis.

Results. None of the devices tested achieved the temperatures set on the dial. The highest temperatures were coronal followed by apical for both devices. The sealers were identical except for the vehicle. The inorganic components included tricalcium silicate, dicalcium silicate and zirconium oxide. No calcium hydroxide was produced by any of the sealers after immersion in physiological solution but calcium was released in solution. The chemistry of both sealers was modified when heated but both recovered when cooled.

Significance. The heat carriers were unreliable and the heat generated inside the canal was not the same as the temperature set on the dial. Since both sealers had identical chemistry save for minimal modifications to the organic component and were both resistant to heat, TotalFill BC sealer is recommended for use with warm vertical compaction technique as it is cheaper and as effective as the HiFlow.

* *Corresponding author.*
E-mail address: J.Camilleri@bham.ac.uk (J. Camilleri).

https://doi.org/10.1016/j.dental.2020.05.008

1. Introduction

The warm vertical compaction of gutta-percha is a very popular obturation technique where the gutta-percha is heated until it changes phase and softens, then it is compacted vertically to take the shape of the prepared root canal [1]. With this technique the obturation is composed mostly of gutta-percha with minimal amounts of sealer. There is no specific sealer that is suggested for use with this technique so all sealer types are believed to be suitable.

The temperature of phase changes to gutta-percha have been reported to be 60 °C to convert from the β form to completely amorphous [2,3]. Regardless of this, all the heat carriers indicate the use of temperature settings ranging from 180 to 230 °C. There seems to be no scientific reason for the use of such temperatures which are unsafe. Increases in temperature of bone has been shown to lead to necrosis as the bone can only tolerate an increase by few degrees [4]. Regardless of the reading on the dial, the temperature of the heat carriers is less [5,6] and the temperature is further dissipated when the heat carrier is placed in a tooth [6,7] and sealer is used in the root canal [6]. As a result of this, the temperature rise on the external surface of the root is minimal [5,6,8].

A rise in temperature in the root canal has deleterious effects on the root canal sealers particularly the epoxy resin-based sealers such as AH Plus (Dentsply). At higher temperatures the AH Plus showed irreversible changes in chemistry [5,6,9,10] and its physical properties were modified with a reduction in the setting time and flow and increase in film thickness [5,6,10–12]. Zinc oxide eugenol-based sealers sustained an increase in setting time when exposed to heat but the flow was unaffected [11] while BioRoot RCS showed changes in its chemistry due to the water evaporation at high temperature [8,10] and iRoot SP had reduced flow [11]. Heating of AH Plus also resulted in an increase in void volume while BioRoot RCS and Gutta-flow were unaffected [12].

Recently a new hydraulic calcium silicate-based sealer which can be used with warm vertical compaction of gutta-percha has been developed. This sealer is claimed to have a similar chemistry to the premixed hydraulic sealers (BC sealers) manufactured by Brasseler/FKG Dentaire but is resistant to heat thus is recommended for use with warm vertical compaction obturation technique. The aim of this research was to determine the temperature generated by two heat carriers inside the root canal in a simulated clinical environment and to characterize both the classical BC sealer and the HiFlow and assess their heat profiles and chemical changes when subjected to high temperatures.

2. Materials and methods

In this study two premixed hydraulic calcium silicate sealers are tested. These included TotalFill and HiFlow BC sealers. Both sealers are manufactured by FKG Dentaire (Chaux-de-Fonds, Switzerland). The HiFlow was developed as an alternative to the BC sealer to be used together with gutta-percha for warm vertical compaction of gutta-percha obturation technique. All analysis was undertaken in triplicate.

2.1. Assessment of heat profiles of the endodontic heat carriers

The heat profiles of two heat carriers used in clincial practice was assessed using a method described in the literature [7]. A single rooted tooth was accessed and chemo-mechanically prepared with ProTaper Gold to a size F2 using sodium hypochlorite irrigation. Three holes 0.8 mm in diameter were drilled along the root face, 2 mm, 8 mm and 12 mm from the apex. K-type thermocouples (0.3 mm diameter, Maplins, UK) were inserted into the root canal through these holes and were held in place by unfilled resin. The thermocouples were connected to a multi-channel data logger (PicoData Logger, TC-08, UK). The whole assembly was placed in alginate to simulate the clinical environment. Temperature was recorded in real-time at a rate of 1 s^{-1} for 30 s using the PicoData logging software. The heat profiles generated by each device were composed of a 10 s pre-heating phase, 4-s heating phase followed by a 10-s cooling phase. Two endodontic heat carriers were tested; E&Q Master (Meta Biomed, Chalfont, PA, USA) and System-B (Orange, CA, USA) both using a fine tip inserted 3 mm short of the apex. The E&Q Master was operated at 180 °C and 230 °C with automatic cut-out after 4 s. System-B was set at 200 °C and a heating cycle of 4 s heating and 10 s cooling was recorded. Each cycle was repeated three times for each heat carrier and set temperature to check for repeatability and the data expressed as mean and standard deviation to the mean.

2.2. Sealer characterization

The organic component of the sealers was extracted in acetone by centrifugation from the unset sealer and analysed by Fourier transform infrared (FT-IR) spectroscopy. The set sealers were characterized by scanning electron microscopy, and X-ray diffraction (XRD) analysis immediately after setting and by XRD after 28 days immersed in Hank's balanced salt solution (HBSS) at 37 °C. The leachate was tested by inductively coupled plasma mass spectroscopy (ICP-OES). All experiments were carried out in triplicate.

The sealers were syringed and specimens 10 mm in diameter and 2 mm thick were made. The specimens were covered by a wet gauze and were placed in an incubator at 37 °C and 100% humidity for 24 h. Setting was verified by indenting the specimen surfaces. One group were tested immediately after setting and the rest were placed in 5 mL of HBSS for 28 days.

For scanning electron microscopy, the specimens were embedded in a cold cure epoxy resin (Epoxy-fix, Struers, Ballerup, Denmark) and the surfaces were polished with an automatic polishing machine (Buhler, Lake Buff, USA) using 250, 500 and 1200 grit diamond discs under water coolant (Struers, Ballerup, Denmark) and polishing cloths (Struers) with ascending grades of diamond-impregnated polishing pastes (Struers). Once polished the embedded specimens were mounted on aluminum stubs and held in place with carbon tape and were sputter coated with gold. The specimens were then viewed under the SEM (Zeiss). The sealers were assessed in back scatter mode to obtain elemental contrast. Images

at different magnifications were obtained. Energy dispersive spectroscopy was performed to assess the elemental distribution.

For the XRD the aged specimens were retrieved from solution and dried in desiccator. For both ages, the sealers were then ground using a mortar and pestle to a fine powder. XRD was performed with a diffractometer (Bruker D8 Advance, Bruker Corp., Billerica, MA, USA) with a CuKa radiation at 40 mA and 45 kV was set to rotate between 10° and 60° with a 0.02° 2θ step and a step time of 0.6 s. Phase identification was undertaken using a search-match software using the ICDD database (International Center for Diffraction Data, Newtown Square, PA, USA). The leachates were assessed for calcium, silicon, zirconium and phosphorus with inductively coupled plasma-optical emission spectrometry (Optima 8000, Perkin Elmer, Waltham, USA). Calculation of results was performed in parts per million (ppm).

2.3. *Investigation of the chemical changes in the sealers*

The sealers were dispensed on a heated diamond plate (MIRacle) attached to a control module (Pike Technologies, USA). The chemical changes to the sealers exposed to a heating and cooling cycle were assessed by FT-IR in ATR mode as described for the sealer characterization. A baseline measurement was taken immediately after sample placement at room temperature (21 ± 1 °C) and then the heating module was activated to heat the samples at a rate of 12.5 °C/min. Measurements were taken from 25 °C to either 50, 100 or 200 °C at increments of 25 °C and then air-cooled to 25 °C and re-measured. All spectra were baseline corrected between 650 and 4000 cm^{-1} and an average (n = 3) was taken. The testing assembly was kept at high relative humidity.

Changes in weight sustained at different temperatures were recorded by thermogravimetric analysis/differential scanning calorimetry, (TGA/DSC; Mettler-Toledo, Beaumont Leys, Leicester, UK). Twenty milligram of set sealers were tested at a temperature range of 50–200 °C at a heating rate of 10 °C min^{-1} and O_2 flow rate of 40 mL min^{-1}. The experiment was repeated three times to ensure repeatability.

The organic component was also extracted after setting at room temperature and after subjecting the sealers to temperatures of 50, 100 and 200 °C and this was tested by FT-IR (n = 3).

2.4. *Statistical analysis*

The statistical analysis was performed using Predictive Analytics Software (PASW) version 18. One-way ANOVA was used to determine whether there were significant differences between data sets. The data was tested to ensure it was normally distributed and then with analysis of variance with P = 0.05 the turkey post hoc test was used.

Table 1 – Temperatures recorded at different levels in the root canal with the various devices and settings.

Device	Temperature (°C)		
	Coronal	Mid-root	Apical
E&Q Master 180	65.3 ± 0.7	43.1 ± 0.2	46.2 ± 0.3
E&Q Master 230	71.0 ± 0.4	45.02 ± 0.3	49.1 ± 0.4
System B 200	59.4 ± 0.9	42.2 ± 0.5	45.4 ± 0.7

Table 2 – Leachate analysis for both sealers showing the concentration of ions leached in solution after 28-day exposure to Hank's balanced salt solution.

Sealer	Ion concentration (ppm)			
	Ca	Zr	Si	P
BC Sealer	867.87	BDL	2.64	−0.22
HiFlow	876.95	BDL	BDL	−0.39

BDL: below detection limits.

3. Results

3.1. *Assessment of heat profiles of endodontic heat carriers*

The heat generated by the endodontic heat carriers is shown in Table 1. The higher setting of the E & Q Master heat carrier led to a higher temperature mostly coronally with minimal changes mid-root and apically. None of the heating devices achieved the temperature set on the dial. All devices exhibited the highest temperature at the coronal portion followed by the apical region with the mid-root exhibiting the lowest temperature. The highest temperature was recorded coronally for the E&Q Master set at 230 °C.

3.2. *Sealer characterization*

The FT-IR scans showing the extraction of the organic component for both sealers is shown in Fig. 1a. The organic component for the sealers was different as it had a different FT-IR signature. The scanning electron micrographs and EDS plots for or both sealers are shown in Fig. 1b. Both sealers had a similar morphology consisting of a composite microstructure with particles of various sizes and opacity. Both materials were composed of calcium, silicon, zirconium and oxygen.

The XRD scans immediately after setting and after 28-day immersion in HBSS are shown in Fig. 1c, and leachate analysis in Table 2. These were identical for both material types. The XRD plots showed predominant peaks of zirconia (ICDD 00-065-0687), tricalcium and dicalcium silicate (ICDD 00-001-1024, 01-070-2450). The leachate analysis (Table 2) showed calcium ion release for both materials with depletion in phosphorus amounts which can account for the taking up of the phosphorus by the materials from the HBSS. The zirconium was below detection limits and low levels of silicon were released.

3.3. *Investigation of the chemical changes in the sealers*

The thermal profiles of the materials while heating to either 50, 100 or 200 °C and cooling are shown in Fig. 2a. The sealers had a different FT-IR signature indicating a difference in chemistry even at room temperature. Both sealers sustained

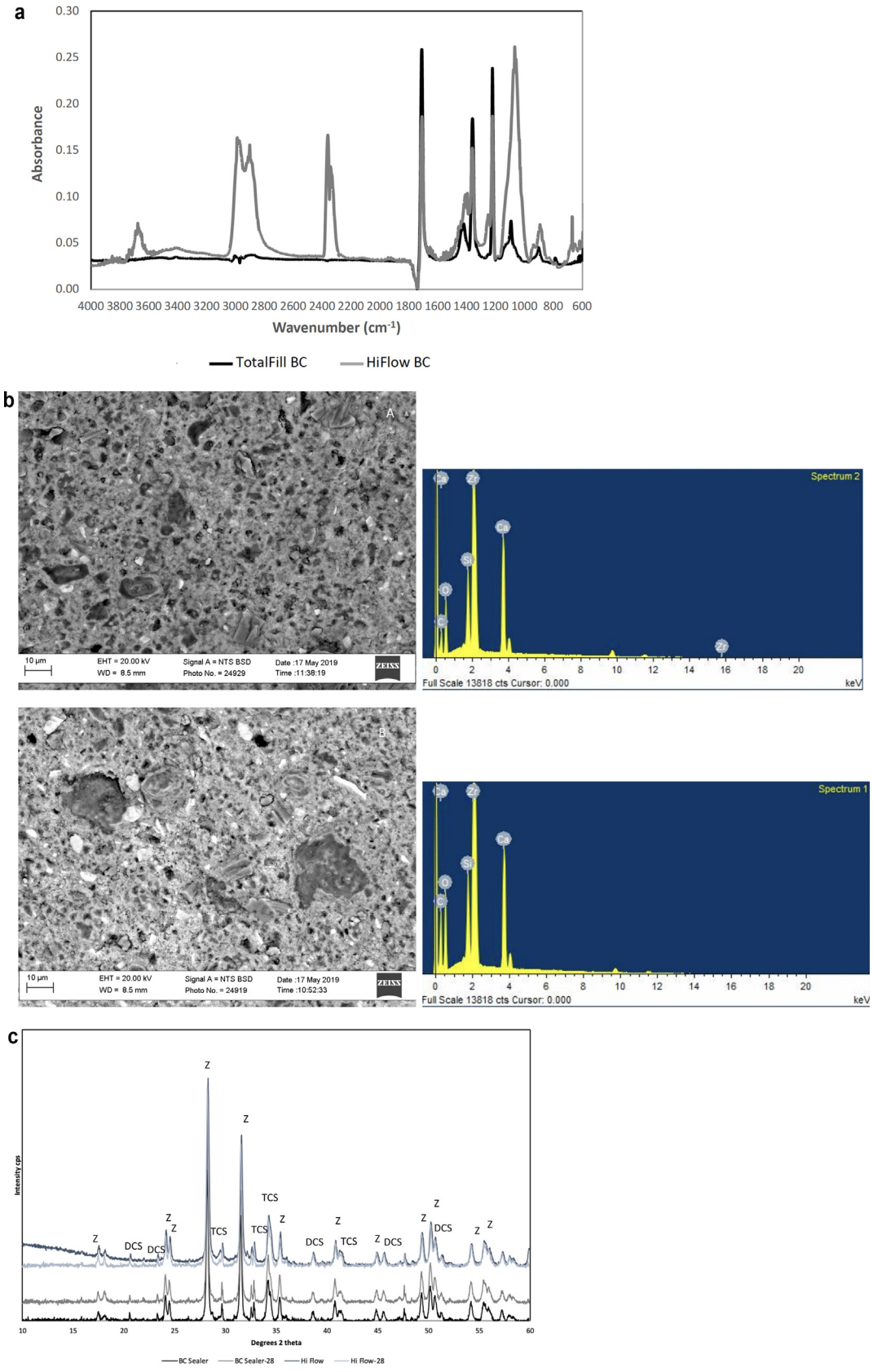

Fig. 1 – (a) FT-IR scan of the organic component extracted from both sealers using acetone and centrifugation. (b) Back scatter scanning electron micrographs and EDS scans of (A) TotalFill BC sealer and (B) HiFlow BC sealer showing microstructure and elemental composition. (c) XRD plots of both sealers immediately after setting and after 28 days immersed in HBSS showing the similar phases present in the materials [DCS: dicalcium silicate; TCS: tricalcium silicate; Z: zirconium oxide].

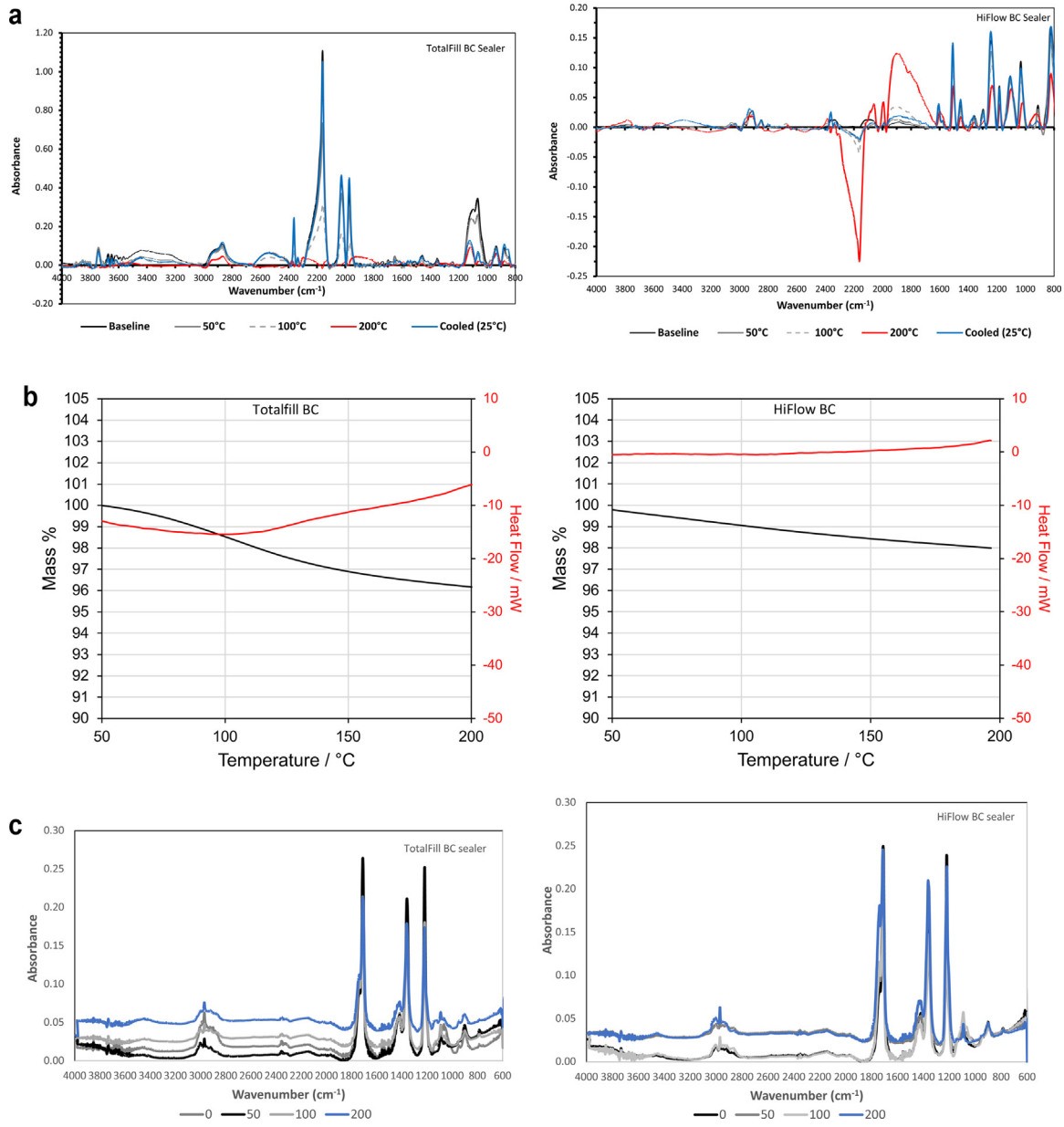

Fig. 2 – (a) FT-IR plots of both sealers exposed to different temperatures showing the chemical changes sustained during heating and cooling. (b) Mass loss (black line) and heat flow (red line) of both sealers calculated by thermographic analysis. (c) FT-IR plots of the organic component of both sealers extracted in acetone after the sealer set at room temperature and after being subjected to different temperatures.

changes in the region of 2200 cm^{-1} which were enhanced the higher the temperature. For both sealers these and the changes at 2000 cm^{-1} for the TotalFill BC and at 1800 cm^{-1} for the HiFlow were reversible.

The TGA data is shown in Fig. 2b. Both sealers showed a mass loss when heated. The mass loss increased with increasing temperatures. Extraction of organic component from set sealers after mixing at room temperature and after being subjected to high temperatures is shown in Fig. 2c. The peaks correspond mainly to the acetone so not much of the vehicle could be extracted after setting and heating.

4. Discussion

The current study investigates two sealer types manufactured by the same company. The TotalFill BC sealer has been characterized previously [13]. HiFlow BC sealer has been launched recently and there is very limited information available for this sealer. All the information regarding its composition is available on the company safety data sheet [14] and the safety data sheet provided is the same for both sealers. The manufacturer recommends the HiFlow for use with warm vertical compaction obturation technique while the Totalfill BC sealer

for single cone obturation technique [15]. The effectiveness of the single cone obturation technique with premixed hydraulic sealers has been verified clinically [16] and a recent survey of clinicians has also endorsed the popularity of this technique [17].

Since HiFlow had not been previously characterized, a full characterization using standard methods such as scanning electron microscopy, energy dispersive spectroscopy and X-ray diffraction analysis was undertaken in conjunction with leachate analysis and extraction of the organic component and analysis. This enabled the identification of the inorganic phases which were shown to be similar to those of Totalfill BC sealer as both sealers were composed mainly of tricalcium silicate and zirconium oxide. [13]. The chemistry of the organic component was not listed on the safety data sheet [14]. The extraction and analysis of the organic components showed a clear difference in the chemistry between the sealers. The sealer chemical similarity has been confirmed in a recent publication [18] which included their identical biological behavior. The only difference was the flow and radiopacity [11,18].

The analysis undertaken showed the material composition rather than the surface chemistry in contact with physiological solution. Apatite nucleation over the materials was not assessed as this phenomenon was shown to be present only in vitro and cannot be translated clinically [19]. All ex vivo analysis has shown the formation of calcium carbonate when hydraulic cements interacted with a clinical environment [20] and even in animal models [21]. A plausible scientific explanation has been reported [22] and the formation of calcium carbonate in vivo has also been recognized for bioglass which is also a bioactive material [23].

In the current research, no calcium hydroxide was observed on setting but calcium ions were released in solution. The lack of formation of calcium hydroxide indicates the slower hydration of this sealer in the early ages which is due to the time required for the organic component to be replaced with reaction by product from the hydration of the tricalcium silicate with water taken up from the surroundings. The limited hydration can be problematic as the calcium ion release by hydraulic cements has been linked to the antimicrobial properties [24]. The use of antimicrobial agents for irrigation has been shown to help with reduction in the microbial load on root canal obturating materials [25]. The biological assessment of both sealers also indicated an initial decreased cell viability which can be correlated to the release of calcium hydroxide which is lower than that of other similar sealer types [13] indicating the limitation of the alternative vehicle in allowing the release of calcium and its crystallization when compared to water-based hydraulic sealers [13].

The heat carriers exhibited a different heat output to the setting on the dial. This has been reported previously [5,6,8,26] and is a cause of concern since the temperature cannot be controlled. In accordance with previous research, the highest temperatures were generated at the coronal aspect of the root canal [5] and the temperatures of the heat carriers recorded inside the root canal ranged from 45 to 70 °C [7].

Based on the temperatures recorded inside the root canal, both sealers were subjected to heat starting at 50 °C which was the temperature recorded in the apical portion where most of the sealer is located during warm vertical compaction of gutta-percha. The evaluations at 100 °C were undertaken to be able to compare to previous studies on sealers [5,6,9–12] and the 200 °C was assessed since the heat carriers were set at this temperature. The temperature applied in the current study was transient to avoid lengthy heat applications which are not clinically valid and also because the frequency and time of heat application effect the sealer chemistry [9]. Both sealers sustained some changes when exposed to heat that intensified with higher temperatures but all changes were reversible indicating that both sealers can be heated even to 200 °C. The Totalfill BC sealer had already been assessed at high temperatures and it showed a similar heat resistance to the current study [26]. The mass loss with increased temperature was minimal and would be stabilized in the long term by the fluid imbibitions in the root canal as the hydraulic sealers interact with the dentin and dentinal fluids in the long term. The unavailability of the organic component to extraction when the sealer had set indicates the setting and the replacement of the organic component with reaction by-product as a result of the hydration initiated by the surrounding liquids.

5. Conclusions

The heat carriers were unreliable and the heat generated inside the canal was not the same as the temperature set on the dial. Both sealers were resistant to heat and had identical chemistries except for modifications to the organic component. TotalFill BC sealer is recommended for use with warm vertical compaction technique as it is cheaper and as effective.

Acknowledgement

Dr Jiangio Liu for his assistance with the testing and Prof Brian Darvell for his guidance with the chemical analysis.

REFERENCES

[1] Schilder H. Filling root canals in three dimensions. Dent Clin North Am 1967:723–44.
[2] Marlin J, Schilder H. Physical properties of gutta-percha when subjected to heat and vertical condensation. Oral Surg Oral Med Oral Pathol 1973;36(6):872–9.
[3] Schilder H, Goodman A, Aldrich W. The thermomechanical properties of gutta-percha. 3. Determination of phase transition temperatures for gutta-percha. Oral Surg Oral Med Oral Pathol 1974;38(1):109–14.
[4] Eriksson AR, Albrektsson T. Temperature threshold levels for heat-induced bone tissue injury: a vital-microscopic study in the rabbit. J Prosthet Dent 1983;50:101–7.
[5] Viapiana R, Baluci CA, Tanomaru-Filho M, Camilleri J. Investigation of chemical changes in sealers during application of the warm vertical compaction technique. Int Endod J 2015;48(1):16–27.
[6] Viapiana R, Guerreiro-Tanomaru JM, Tanomaru-Filho M, Camilleri J. Investigation of the effect of sealer use on the heat generated at the external root surface during root canal obturation using warm vertical compaction technique with System B heat source. J Endod 2014;40(4):555–61.

[7] Donnermeyer D, Schäfer E, Bürklein S. Real-time intracanal temperature measurement during different obturation techniques. J Endod 2018;44(12):1832–6.
[8] Venturi M, Pasquantonio G, Falconi M, Breschi L. Temperature change within gutta-percha induced by the System-B Heat Source. Int Endod J 2002;35(9):740–6.
[9] Atmeh AR, AlShwaimi E. The effect of heating time and temperature on epoxy resin and calcium silicate-based endodontic sealers. J Endod 2017;43(12):2112–8.
[10] Camilleri J. Sealers and warm gutta-percha obturation techniques. J Endod 2015;41(1):72–8.
[11] Qu W, Bai W, Liang YH, Gao XJ. Influence of warm vertical compaction technique on physical properties of root canal sealers. J Endod 2016;42(12):1829–33.
[12] Heran J, Khalid S, Albaaj F, Tomson PL, Camilleri J. The single cone obturation technique with a modified warm filler. J Dent 2019;89:103181.
[13] Xuereb M, Vella P, Damidot D, Sammut CV, Camilleri J. In situ assessment of the setting of tricalcium silicate-based sealers using a dentin pressure model. J Endod 2015;41(1):111–24.
[14] https://www.fkg.ch/sites/default/files/FKG_TotalFill%20BC%20Sealer_Safety%20data%20sheet.pdf.
[15] https://brasselerusadental.com/products/endosequence-bc-sealer-hiflow/.
[16] Chybowski EA, Glickman GN, Patel Y, Fleury A, Solomon E, He J. Clinical outcome of non-surgical root canal treatment using a single-cone technique with endosequence bioceramic sealer: a retrospective analysis. J Endod 2018;44(6):941–5.
[17] Guivarc'h M, Jeanneau C, Giraud T, Pommel L, About I, Azim AA, et al. An international survey on the use of calcium silicate-based sealers in non-surgical endodontic treatment. Clin Oral Investig 2020;24(1):417–24.
[18] Chen B, Haapasalo M, Mobuchon C, Li X, Ma J, Shen Y. Cytotoxicity and the effect of temperature on physical properties and chemical composition of a new calcium silicate-based root canal sealer. J Endod 2020;46(4):531–8.
[19] Meschi N, Li X, Van Gorp G, Camilleri J, Van Meerbeek B, Lambrechts P. Bioactivity potential of Portland cement in regenerative endodontic procedures: from clinic to lab. Dent Mater 2019;35(9):1342–50.
[20] Moinzadeh AT, Aznar Portoles C, Schembri Wismayer P, Camilleri J. Bioactivity potential of endosequence BC RRM putty. J Endod 2016;42(4):615–21.
[21] Schembri Wismayer P, Lung CY, Rappa F, Cappello F, Camilleri J. Assessment of the interaction of Portland cement-based materials with blood and tissue fluids using an animal model. Sci Rep 2016;29(6):34547, http://dx.doi.org/10.1038/srep34547.
[22] Bohner M, Lemaitre J. Can bioactivity be tested in vitro with SBF solution? Biomaterials 2009;30:2175–9.
[23] Mozafari M, Banijamali S, Baino F, Kargozar S, Hill RG. Calcium carbonate: adored and ignored in bioactivity assessment. Acta Biomater 2019;91:35–47.
[24] Koutroulis A, Kuehne SA, Cooper PR, Camilleri J. The role of calcium ion release on biocompatibility and antimicrobial properties of hydraulic cements. Sci Rep 2019;9(13):19019, http://dx.doi.org/10.1038/s41598-019-55288-3.
[25] Xu J, He J, Shen Y, Zhou X, Huang D, Gao Y, et al. Influence of endodontic procedure on the adherence of *Enterococcus faecalis*. J Endod 2019;45:943–9.
[26] Atmeh AR, Hadis M, Camilleri J. Real-time chemical analysis of root filling materials with heating: guidelines for safe temperature levels. Int Endod J 2020, http://dx.doi.org/10.1111/iej.13269 [Epub ahead of print].